Analysis of Lysergic Acid Diethylamide (LSD)

1. **Background**
2. **Objective**
3. **Scope**
4. **Responsibility**
5. **Related Documents**
6. **Definitions**
7. **Supplies, Equipment & Reagents**
8. **Safety**
9. **Reagent Preparation**
10. **Procedure**
11. **Documentation**
12. **Attachment**

1. Background

Lysergic acid diethylamide (LSD), commonly referred to as "acid", is a synthetic hallucinogen. LSD is manufactured from lysergic acid, which is found in ergot, a fungus that grows on rye and other grains. It is a colorless, odorless, and tasteless liquid. It comes in a variety of forms, but is always taken orally. LSD is most commonly found of small squares of paper called blotter (full sheet of paper are decorated with artwork or designs, perforated, then soaked in liquid solution and dried). Other forms include, tablets (microdots), gelatin squares (window panes), liquid, liquid sugar cubes and powder. Additionally, LSD has been embedded in candy such as "Gummy Worms," "Sweet Tarts," "Smarties," and "Pez."

2. Objective

The objective of this SOP is to establish guidelines to be used for the analysis of a sample that may contain lysergic acid diethylamide (LSD).

3. Scope

This SOP is to be used by the laboratory staff of the Division of Analytical Chemistry at William A. Hinton State Laboratory Institute in Boston, MA.

4. Responsibility

Chemists are responsible for acquiring glassware, preparing chemical reagents and standards, sample analysis, and reporting. Chemists also perform instrument calibrations, maintenance and troubleshooting, ordering of supplies and other necessary tasks related to this analysis.

Laboratory Supervisors ensure that chemists are following this SOP. They may perform the duties of the chemists and must review raw data and reports generated by chemists. The Supervisor may advise the chemists of alternative testing methods. They ensure that quality control measures are within acceptable limits and determine when corrective actions are needed. They coordinate proficiency testing (PT), reporting and distribution of PT results. They oversee sample results distribution to outside agencies.

Directors ensure that the SOP is being followed and reviewed on a regular basis. They provide approval of standard operating procedures and review quality control documentations.

1. Related Documents

Cole, Michael, "The Analysis of Controlled Substances," London: John Wiley & Sons Ltd., 2003
Drug Enforcement Administration, "Basic Training Program for Forensic Drug Chemists," Drug Enforcement Administration.
Mills III, Terry et al, "Instrumental Data for Drug Analysis," 3rd ed., 6 vols., New York: CRC Press, 2006.
Moffat, A.C. et al, "Clarke's Isolation and Identification of Drugs," 2nd ed., London: The Pharmaceutical Press, 1986.
Moffat, A.C. et al. "Clarke's Analysis of Drugs and Poisons," 3rd ed., London: The Pharmaceutical Press, 2004.
Saferstein, Richard, "Forensic Science Handbook," New Jersey: Prentice Hall, 1988.
Scientific Working Group for the Analysis of Seized Drug Recommendation, 6th ed., "Part III A & B, Methods of Analysis/Sampling of Seized Drug for Qualitative Analysis," July 2011

6. Definitions

GC: Gas Chromatography
GC/MS: Gas Chromatography/Mass Spectrometry

7. Supplies, Equipment & Reagents

Supplies

Culture tubes
Scissors
Spatula
Pasteur pipette
Tweezers
Volumetric Flask
Weighing dish
Weighing paper
GC vials with Teflon caps

Equipment

Analytical Balance

Ultraviolet (UV) Lamp
GC with FID
GC/MS

Reagents

p-dimethylaminobenzaldehyde (p-DMAB)
Lysergic acid diethylamide (LSD)
Lysergic acid methylpropylamide (LAMPA)
95% Ethanol
Concentrated Hydrochloric Acid
Methanol
Cocaine Hydrochloride
Codeine Phosphate
Chloroform
Sodium Bicarbonate
Tartaric Acid
Water

8. Safety

Due to the potential hazards, appropriate precautions should be taken as necessary. This includes, but is not limited to, the use of fume hoods, gloves, masks and safety glasses. Lab coats are to be worn at all times in the unit, unless performing administrative duties.

9. Reagent Preparation

Ehrlich's Reagent
Dissolve 2.5g of p-dimethylaminobenzaldehyde and bring to volume with 50mL of 95% Ethanol. Mix the solution until completely dissolved. Solution must be protected from light.

Lysergic Acid Diethylamide (LSD) Standard
Dissolve 2.0mg of lysergic acid diethylamide and bring to volume with 1.8mL of methanol. Mix the solution until completely dissolved.

Lysergic acid methylpropylamide (LAMPA) Standard
Dissolve 2.0mg of lysergic acid methyl propyl amide and bring to volume with 1.8mL of methanol. Mix the solution until completely dissolved.

Cocaine/Codeine Standard (QC Mix)
Dissolve 10.0 mg of cocaine hydrochloride and 10.0mg of codeine phosphate and bring to volume with 10mL of methanol. Mix the solution until completely dissolved.

10. Procedure

A. Document observations on the Drug Analysis Form noting the number, type (e.g. liquid, paper squares, window panes, sugar cubes, etc) and marking of all items.
B. Sampling Plan
    i.
C. Color Test

i. Cut the suspected paper into small pieces or take a small amount of powder or substance and place into a labeled culture tube.
ii. Add 1-3 drops of Ehrlich's Reagent to the sample and agitate the culture tube.
iii. Add 1-3 drops of Concentrated Hydrochloric Acid to the sample and agitate the culture tube.
iv. A positive reaction to the Ehrlich's Reagent Color test is a blue/purple color. The results will be recorded on the Drug Analysis Form by documenting the actual color/s observed. Negative observations will be recorded by stating no reaction present or no color change.

D. Ultraviolet Fluorescence Test
i. Cut the suspected paper into small pieces or tale a small amount of powder or substance and place into a labeled culture tube.
ii. Dilute sample with methanol until it just covers the sample.
iii. Vortex the culture tube and let stand for 1 day.
iv. Remove the methanolic extract and place into a labeled GC vial and cap tightly.
v. Place a 1-2 drops the methanolic extract onto filter paper and allow to dry.
vi. Observe the filter paper under long wavelength UV light (360 nm).
vii. If positive, LSD may fluoresce a blue/purple color. The results will be recorded on the Drug Analysis Form by documenting the actual color/s observed. Negative observations will be recorded by stating no reaction present.

E. Extraction
i. LSD may be dry extracted with methanol from blotter paper and other matrices.
ii. LSD can be extracted from basic aqueous solution with organic solvents.
iii. To extract LSD from sample like plastic, it may be necessary to dissolve the sample in a methanol/chloroform mixture.
iv. If samples are in a matrix which is impervious to organic solvents, LSD may be extracted by creating the tartrate salt, followed by a base extraction.

F. Gas Chromatography
i. The extract from section (E) can be used for the GC analysis. Place the extract into a labeled GC vial and cap tightly.
ii. Initiate auto sampler sequence using the GENSCAN method running a blank solvent between each unknown sample and reference standard/s.
iii. Compare retention time of the each sample with the reference standard/s. Also check the chromatograph to determine if the sample needs to be diluted or concentrated.
iv. Positive GC analysis will be recorded on the Drug Analysis Form by the use of a plus (+). The result is considered positive when the retention time of the sample and the reference standard (Cocaine/Codeine Mix) meet the laboratory criteria and are specified in the notes. Negative observations will be recorded by the use of a negative (-).

G. Gas Chromatography/Mass Spectrometry
i. Perform a preventative maintenance on the instrument by cleaning the injection port and changing the liner.
ii. Confirmatory analysis can be performed using the GC vial from the previous section (E).
iii. Initiate auto sampler sequence using the LSD method running a blank solvent between each unknown sample and reference standard/s.
iv. Compare retention time and ion spectra of the each sample with the reference standard/s (LSD & LAMPA).
v. Document the date analyzed and results of the GC/MS onto the MS Tracking Sheet, Drug Analysis Form and Control Card.

11. Documentation

A. All results will be documented on the Drug Analysis Form.
B. All raw data will be generated and filed according to the laboratory policy.
C. A certificate of analysis will be generated for each lab number which will document the results.

12. Attachments

GC Method

GC/MS Method